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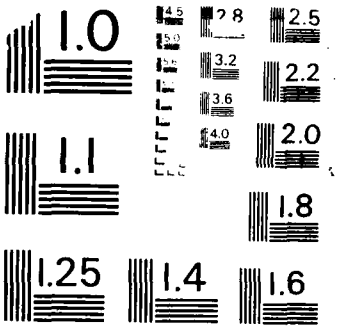

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Interfacial structure property relationships at the fiber matrix interphase in advanced composite materials. This grant, approved under the DOD University Research Instrumentation Grant Program was used to partially fund the purchase of surface spectroscopy equipment. The report contains the listings and descriptions of equipment actually acquired, a summary of research projects on which the equipment will be used, and other research work of interest to DOD for which this equipment will be used.

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AFOSR-TR- 87-1780

October 16, 1987

Air Force Office of Scientific Research
AFOSR/PKD (Lisa Ray)
Bolling Air Force Base
Washington, D.C. 20332-6448

Dear Ms. Ray:

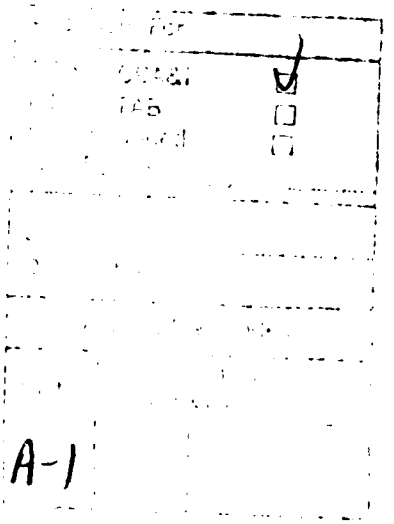
Attached is the final report for AFOSR Grant No. AFOSR-86-0286 entitled,"INTERFACIAL STRUCTURE-PROPERTY RELATIONSHIPS AT THE FIBER MATRIX INTERPHASE IN ADVANCED COMPOSITE MATERIALS". This grant, approved under the DOD University Research Instrumentation Grant Program was used to partially fund the purchase of surface spectroscopy equipment. The report contains the listing and description of equipment actually acquired, a summary of research projects on which the equipment will be used, and other research work of interest to DOD for which this equipment will be used.

Yours truly,

Lawrence T. Drzal

Lawrence T. Drzal
Professor of Chemical Engineering

cc: AFOSR/NE J. Hager
S. M. Luzkow, Contract and Grant Administration



FINAL REPORT

Air Force Office of Scientific Research

Grant No. AFOSR-86-0286
MSU Account No. 71-2314

University Research Instrumentation Program

INTERFACIAL STRUCTURE-PROPERTY RELATIONSHIPS AT THE FIBER-MATRIX INTERPHASE IN ADVANCED COMPOSITES

Principal Investigator:

Professor Lawrence T. Drzal

Department of Chemical Engineering
Composite Materials and Structures Center
Michigan State University
East Lansing, Michigan 48824-1326
517-353-7759

Research Title:

INTERFACIAL STRUCTURE-PROPERTY RELATIONSHIPS AT THE FIBER MATRIX INTERPHASE IN ADVANCED COMPOSITE MATERIALS

Principal Investigator:

Professor Lawrence T. Drzal

Part I. Research Equipment Purchased:

Item No. 1 Scanning Auger Microprobe Spectrometer

Source: The Perkin-Elmer Company
Physical Electronics Division
6509 Flying Cloud Drive
Eden Prarie, Minnesota 55344

Model: 660 Scanning Auger Microprobe

Item No. 2 Small Spot X-ray Photoelectron Spectrometer

Source: The Perkin-Elmer Company
Physical Electronics Division
6509 Flying Cloud Drive
Eden Prarie, Minnesota 55344

Model: 5400 Small Spot X-ray Photoelectron Spectrometer

Costs and Finances:

Scanning Auger Spectrometer	$409,950
X-ray Photoelectron Spectrometer	$440,570
TOTAL	$850,520
Perkin-Elmer Contribution	$272,927
AFOSR Grant No. 86-0286	$230,000
MSU	$347.593
TOTAL	$850,000

Special Circumstances:

The use of surface spectroscopic research instrumentation requires a clean controlled temperature and humidity environment as well as close proximity to the material to be analyzed. As part of the receipt of this grant The College of Engineering's Composite Materials and Structures Center has

provided approximately 1000 square feet of air conditioned laboratory space within the Center for this equipment. (The value of this piece computed at $100 per square foot is $100,000.) Researchers in polymeric, metallic, cementitious and ceramic matrix composite materials will be conducting their research activities in the Center and will be involved in the use of the surface spectrometer equipment. The dedicated space is part of the Engineering Research Complex which was made available for occupancy in August of 1987.

Michigan State University has recognized the value of this research equipment and has funded additional accessories to make the data collection, experimental variables and experimental options for this equipment wider in scope than that originally purchased. MSU has added funds totaling $48,763 for the purchase of heating and cooling accessories, multiple sample mounting, and automatic data collection.

The Perkin-Elmer Corporation has generously made available to MSU a one-time University Research Equipment Purchase Cost Reduction for the purchase of these spectrometers amounting to approximately 50% for the X-ray Photoelectron Spectrometer and 25% for the Scanning Auger Microprobe.

The spectrometers were received from the Perkin-Elmer Corporation in April of 1988. Installation was not started until the internal construction of the research space in the Engineering Research Complex was completed. Installation and acceptance of the spectrometers was completed in September of 1988. Coincidently a search for a full-time surface spectroscopist to operate the instruments has been initiated with selection and hiring of the individual to be completed by November of 1988.

Part II. Research Projects on which the equipment will be used:

a. "Interaction of Microwave Energy with the Fiber-Matrix Interphase in Composite Materials", L. T. Drzal, P.I. Sponsor: DARPA. Microwave energy is being investigated as a curing medium for advanced composites. The interaction with both the fiber surface and the polymer near the fiber surface in the interphase can be affected differently by electromagnetic radiation than by thermal energy. This project seeks to understand the interactions, maximize the adhesion and identify the operating mechanisms responsible. Surface spectroscopy will be used to quantify the polymer fiber chemical interactions and changes in fiber surface chemistry with radiation dosage.

b. "Study of the Interface/Interphase in Thick Section Composites", L. T. Drzal, P.I. Sponsor: ONR, U.Ill (University Research Initiative). The events which occur in the processing of advanced composite materials have a large effect on the final state of the cured composite. Thick section composites in particular are especially sensitive to processing variations which can result in internal defects. This research program is directed at developing a molecular understanding of the development and changes in

interfacial properties of advanced composite materials with processing in thermal and microwave environments. Surface spectroscopy will be used to identify the chemical reactions and chemical changes which occur at the fiber surface and the fiber-matrix interphase during the processing of advanced composite materials.

c. "Fiber-Matrix Adhesion and Its Relation to Composite Properties". L. T. Drzal, P.I. Sponsor: ONR. New high strain carbon fibers and tougher thermoset and thermoplastic matrices require optimal surface treatments to insure maximum translation of fiber and matrix properties into the composite. This requires optimum levels of adhesion. At the present time the fundamental mechanisms of adhesion are not known. This program is directed at developing structure property relationships between fiber-matrix adhesion and composite mechanical properties. Surface spectroscopic analysis will be used to characterize the surface atomic and molecular structure of advanced carbon fiber reinforcements as well as their reaction to thermoset and thermoplastic matrices used with these materials. Mechanical property measurements will be made to relate adhesion levels to composite properties.

d. "High Temperature Metal-Matrix and Ceramic Matrix Composites" G. Gottstein and E. Case, P.I. This research is directed at the processing and mechanical property measurement of nickel aluminides reinforced with silicon carbide fibers. Surface spectroscopy will be used to characterize interfacial reaction between matrix and fiber as well as failure locus after mechanical and environmental testing.

Part 3. Other Research Work of interest to DOD.

a. "The Interphase in Fiber Reinforced Concrete." P. Soroushian, P.I. This project is actively pursuing the reinforcement of cementitious materials with carbon and other reinforcing fibers. Current results indicate that small amounts of reinforcement of carbon fiber produce large improvements in composite properties. An effort to optimize and understand the role of fiber-matrix adhesion is being conducted within this program. The molecular level interfacial interactions between fiber and cement as well as cementitious additives will be completed with surface spectroscopic techniques.

b. "Fundamental Investigation of the Kevlar-49/Polymer Interphase" L. T. Drzal, P.I., Sponsor: E.I. duPont. Aramid fibers have poor adhesion to thermoset matrices compared to inorganic fibers. Attempts to improve this adhesion level have been unsuccessful. This project seeks to establish the molecular state of the aramid fiber surface and to alter the fiber surface chemistry for optimum adhesion to thermoset matrices.

c. "Characterization of the Glass Fiber-Epoxy Interphase in Composite Materials", L. T. Drzal, P.I. Sponsor: Dow Chemical Company. An in-situ measurement technique for the measuring fiber-matrix adhesion is a

requirement for research as well as product reliability. This program seeks to establish the relationship between molecular level events as measured by the embedded single fiber measurement technique with a macroscopic measurement of the interfacial shear strength as conducted by the fiber end compression technique. Surface spectroscopy of glass fiber surfaces and interphases formed with epoxy matrices will be a central area of investigation of this project.

d. "Compressive Strength Improvement of Polymeric Reinforcing Fibers" L. T. Drzal, P.I. Sponsor: AFWAL/Dow Chemical Company. This research subcontract with the Dow Chemical Company under an Air Force Contract seeks to improve the level of adhesion between polymeric reinforcing fibers and thermoset and thermoplastic matrices in respect to their effect on the compressive properties of their composites. Surface spectroscopy will be used to determine the chemical and atomic state of the fiber surface and to monitor its change with surface treatment.

e. "Characterization of the Interfacial Properties of Polyphenylene Sulfide (PPS) Polymers" L. T. Drzal, P.I., Sponsor: Phillips Petroleum. Polyphenylene Sulfide is a tough, high temperature polymer suitable for use as a matrix material in advanced composites. The interfacial properties of composites made with this matrix and carbon fibers has not been optimized for mechanical properties. This investigation will determine the molecular structure of the PPS matrix and how this molecular and morphological structure changes with proximity to the reinforcing fiber surface and chemistry. Surface spectroscopy will be used to assess these chemical and atomic changes.

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